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NASA TECH BRIEF

Ames Research Center

Free-Radical Solution-Polymerization of Trifluoronitrosomethane with Tetrafluoroethylene

The effect of including free-radical initiators in solution-polymerizations of trifluoronitrosomethane (CF_3NO) with tetrafluoroethylene (C_2F_4) was investigated to determine if more than an equimolar quantity of C_2F_4 could be incorporated in the copolymer.

The polymerization initiators selected for study were methylethyl ketone peroxide, *t*-butyl hydroperoxide, and isopropylperoxydicarbonate; methylene chloride was selected as solvent and the temperature schedule for the polymerization was set by preliminary experiments at 5 days at −35°C and 2 days at 0°C. The free-radical initiator was used at the rate of 0.1 g for each 0.20 mole of CF_3NO. In each case, a 50-percent excess of C_2F_4 was charged into the reaction vessel.

Polymer conversions were good, and in some instances experimental data suggested that the C_2F_4 content exceeded the theoretical amount for a 1 : 1 copolymer. The products were elastomeric gums which exhibited a very low order of flow; however, they were not completely soluble in the FC-43 solvent used for measurement of relative viscosity. Hexafluorobenzene also did not dissolve the copolymer completely.

For polymerization, a 500-ml heavy-walled glass reactor (equipped with aerosol-compatible couplings and needle valve) was charged with 100 g methylene chloride, followed by 0.1 g of the free-radical initiator. The solvent was degassed three times by a freeze-thaw technique at liquid nitrogen temperature. Then 20.0 g (0.2 mole) CF_3NO and 32.0 g (0.3 mole) C_2F_4 were charged successively into the polymerization vessel while it was immersed in liquid nitrogen. Transfers were made by expansion of the vaporized monomers into a calibrated volume at a measured pressure followed by liquefaction at liquid nitrogen temperature. The sealed vessel (shaken periodically) was maintained in a refrigerated bath at −35°C for a 5-day period and then for 2 days at 0°C. The cooled vessel was vented and allowed to come to room temperature while its contents were kept under a nitrogen atmosphere. The system was evacuated to remove residual monomer, solvent, and other volatiles. The remaining gum was heated for 4 hours at 60°C in nitrogen at low pressure.

Note:

Requests for further information may be directed to:
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Patent status:

No patent action is contemplated by NASA.

Source: Samuel A. Glickman of
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